

# National Bureau of Standards

## TECHNICAL NEWS BULLETIN

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### Automatic Ionosphere Recorder

A new instrument for automatic recording of ionospheric phenomena, which are of great practical importance in radio propagation, is now in operation at the Bureau's ionospheric research station at Sterling, Virginia. The prototype model of the recorder, developed by the Bureau's Central Radio Propagation Laboratory, was completed in time to participate in the recent Army Air Forces—National Geographic Society Eclipse Expedition in Brazil. In addition to special records during the May 20 eclipse of the sun, valuable data were obtained on general ionosphere conditions. Plans are being made to install these recorders in all the ionosphere stations operated by the Bureau.

Long-distance radio transmission would be impossible if it were not for the ionosphere, a series of ionized layers in the atmosphere 50 to 250 miles above the earth that reflect radio waves back to earth. The new recorder will provide automatic and continuous measurements of the heights of the various layers and of critical frequency, that is, the maximum frequency that is reflected back to earth rather than passing off into space.

The model C2 recorder was designed to utilize the so-called multifrequency technic of investigating the layers of the ionosphere. It is the first completely automatic recorder to use, in continuous heavy duty, the heterodyne pulse transmitter arrangement recently described by P. G. Sulzer.<sup>1</sup> In this arrangement the entire frequency range from 1.0 to 25.0 megacycles per second is covered continuously without switching bands. The receiver is tuned with the transmitter throughout its frequency range. Pulse transmissions are used simi-

lar to those employed in radar except with varying probing frequency. The frequency is plotted against the time delay of the echoes from the ionosphere, an interval that corresponds to twice the height of reflection. One sweep in frequency from lower to upper limits produces each ionosphere record in a time interval of as little as  $7\frac{1}{2}$  seconds.

Two oscilloscopes provide indications corresponding in essential detail to the radar "A" and radar "B" scans. A special 35-millimeter camera, driven by the motor that operates the transmitter oscillator, makes a continuous film record of the indications of the main oscilloscope. As the image of the sweep line is oriented at right angles to the direction of film travel, the graph of height reflection (time delay) versus frequency is recorded on height scales of 500, 1,000, and 4,000 kilometers.

A standard 16-millimeter motion picture camera is provided to photograph the monitoring oscilloscope. When a series of 16-millimeter film records, made at  $7\frac{1}{2}$ - or 15-second intervals over a long period of time, are projected, a striking accelerated version of the changes taking place in the ionosphere is observed.

The main cabinet of the new ionosphere recorder houses no moving parts, but encloses the transmitter mixer, transmitter wide-band amplifier, receiver mixer, receiver intermediate frequency unit with detector and video stages, pulsing and keying circuits for the transmitter, the transmitter fixed frequency unit, and all the power supplies for these units.

A smaller cabinet on rollers contains the recording and monitoring oscilloscopes, the recording 35-milli-

<sup>1</sup> Ionosphere measuring equipment, *Electronics* 19, 137 (July 1946).

meter camera, the variable frequency oscillator, the camera and oscillator drive motor, switches and their associated control, time base and sweep-generating circuits, and the necessary power supplies.

Other features of the model C2 recorder are: A pulse receiver having large dynamic range and a differentiating circuit to minimize interference by CW (continuous wave) and the broadcast stations, provision for automatic operation by a clock completely independent of power-line frequency, the use of hermetically sealed units to insure reliable operation in many climates, and regulation of every important direct-current voltage.

The replacement of the old recording units in the ionosphere station network with an automatic and continuous recorder will assure a more comprehensive and reliable flow of worldwide ionosphere data into the National Bureau of Standards. To date, the principal emphasis in the study of ionospheric phenomena has been the practical use of the information in the prediction of radio propagation conditions. However, data on the ionosphere, which may be considered as a strategically located astrophysical radiation laboratory, will reveal more and more of the characteristics of radiation from the sun, and the physical conditions of the outer atmosphere, its temperatures and densities, mean free paths of electrons, recombination processes, and geomagnetic effects. These data may also lead to information on air circulation and other phenomena in the lower atmosphere (stratosphere and troposphere).



The new ionosphere recorder developed by the Central Radio Propagation Laboratory automatically records ionosphere fluctuations, which are of great practical importance in radio propagation predictions. The main cabinet on the right houses the transmitter and the receiver. The indicator-recorder on the left provides visual observation on a monitoring oscilloscope simultaneously with a 35-millimeter recording of the main oscilloscope.



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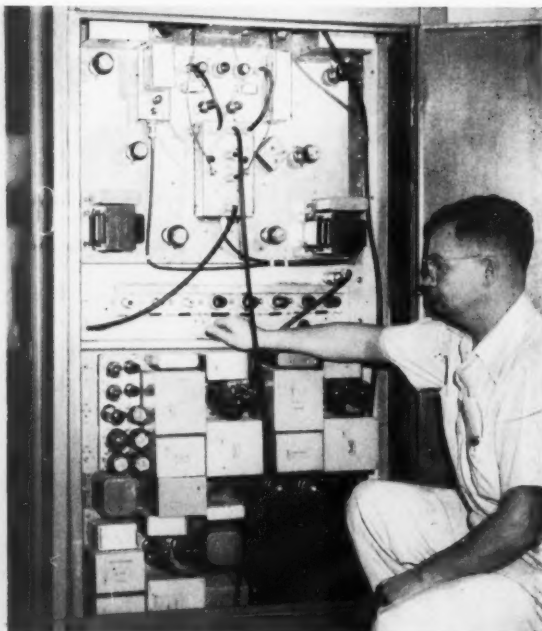
U. S. DEPARTMENT OF COMMERCE  
W. AVERELL HARRIMAN, *Secretary*  
NATIONAL BUREAU OF STANDARDS  
E. U. Condon, *Director*

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# Electroless Plating on Metals

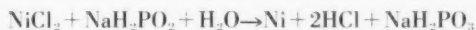
## by Chemical Reduction

A new process for plating nickel and cobalt on metal surfaces without the use of electric current, developed by Abner Brenner and Grace E. Riddell of the Bureau's electroplating laboratory, is brought about by chemical reduction of a nickel or cobalt salt with hypophosphite in hot solution. The reaction is catalytic, and under the prescribed conditions of concentration and pH, no plating occurs unless certain metals, such as steel or nickel, are introduced in the bath. The reduction then occurs only on the surface of the immersed metal with the production of an adherent coating of 93 to 97 percent purity.

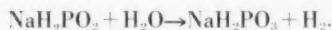
Photomicrographs of deposits obtained in this way show both a laminar and a columnar structure, similar to bright nickel electrodeposits. The electroless deposits are of good quality—sound though brittle, and usually bright. As they can be made as hard as tool steel, the method may prove useful where hard, wear-resistant surfaces are required, as in bearings. The process is particularly applicable to the plating of recesses, irregular-shaped objects, and enclosed areas such as tubes, where a centered internal electrode with special current leads would be needed in electroplating.

The equipment is simple and more easily assembled than that required for electroplating. No generators, rheostats, special racks, or contacts are necessary. Small parts that cannot be barrel-plated economically are readily plated by the electroless process if suspended by a string or in a bag affording ample exposure of the metal surface to the solution. There is no need of constant motion, as in barrel plating, as current distribution is not involved.

Although electroless deposits of cobalt and cobalt-nickel alloys have been obtained only from ammoniacal solutions, nickel can be deposited from either acid or alkaline solutions. The reactions, requiring a temperature about 90° C. are given in the following equations (in which cobalt may also be written for nickel):



or



The first reaction is the important one, resulting in the deposition of nickel; the second reaction tends to lower the efficiency of the process through oxidation of the hypophosphite.

A unique feature of the electroless process is the catalytic initiation of the reaction by the following metals: Iron or steel, nickel, gold, cobalt, palladium, and aluminum. Unless one of these metals is introduced into the solution, no reaction takes place. Once started, the reaction continues at the metallic surface and only rarely occurs in other parts of the bath. For

*Suggested compositions for the various electroless solutions*

	Nickel		Cobalt	Cobalt-Nickel
	Alkaline	Acid		
	g/liter	g/liter	g/liter	g/liter
Nickel chloride, $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	30	30		30
Cobalt chloride, $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$			30	20
Sodium hypophosphite, $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$	10	10	20	20
Sodium citrate, $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 5\text{H}_2\text{O}$	100		35	
Rochelle salt, $\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$				200
Sodium hydroxyacetate, $\text{NaC}_2\text{H}_3\text{O}_2$		50		
Ammonium chloride, $\text{NH}_4\text{Cl}$	50		50	50
Alkali for neutralizing pH	$\text{NH}_4\text{OH}$ 8 to 10	$\text{NaOH}$ 4 to 6	$\text{NH}_4\text{OH}$ 9 to 10	$\text{NH}_4\text{OH}$ 8 to 10
Rate of deposition, millimeter/hour	0.008	0.015	0.015	0.015
Rate of deposition, inch/hour	.0003	.0006	.0006	.0006
Appearance of deposit	Bright	Semi-bright	Dull	Semi-bright

this reason, the containing vessel should be of glass, plastic, or other noncatalytic material.

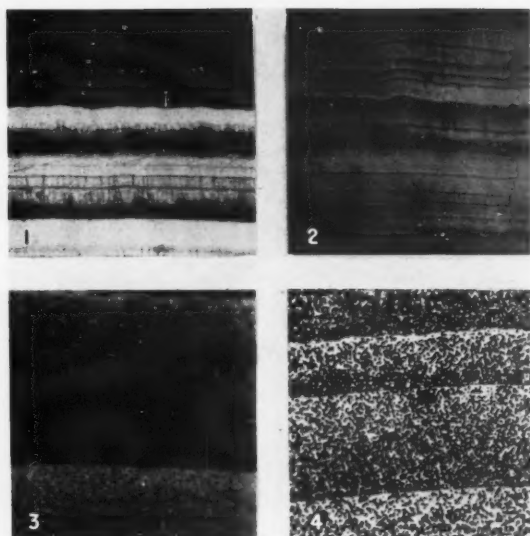
Objects to be plated are cleaned by any of the accepted procedures and are given an acid dip before being suspended in the hot solution. The rate of deposition is about the same as in barrel electroplating, ranging from 0.0002 to 0.0008 inch per hour, depending upon the type of solution used. During the process, the pH must be kept within a certain range, and if the operation is of long duration, the nickel salt and hypophosphite are replenished at regular intervals.

The composition of the plating bath may vary within rather wide limits. Suggested solutions are given in the table. In addition to the metal ions and hypophosphite, the alkaline solutions contain hydroxycarboxylic salts and ammonium salts to prevent precipitation of the metal salts, and certain alkalies to regulate the pH.

Electroless plating on noncatalytic metal surfaces may be accomplished in two ways. If a film of palladium or rhodium of nearly monatomic thickness is first applied by chemical replacement on a noncatalytic metal, deposition of nickel or cobalt will occur on the activated surface. Electroless plating of copper may be carried out in this way. A second method of initiating the reduction is to bring a less noble metal, such as iron or aluminum, in contact with the noncatalytic metal while it is immersed in the hot electroless solution. Once the process has been started, it continues because of the catalytic action of the initial deposit.

As formed, the electroless nickel deposits are brittle but become ductile when heated. These deposits are harder than the ordinary electrodeposited nickel, and upon heating their hardness is still further increased. This is in contrast to the behavior of "hard" electrodeposited nickel, which has high initial hardness but softens upon heating. The hardening may be explained as "precipitation hardening", probably of phosphides.

The adhesion of the nickel deposit to mild steel is such that it cannot be flaked off by bending, but on high-



Photomicrographs show precipitation hardening of "electroless" nickel deposits. (1) As deposited; hardness 500 (Knoop scale). (2,3,4) After annealing for 30 minutes at 400°, 600°, and 800° C, respectively. Note excessive precipitation in (4). Hardness in (2) and (4) is 800 and 470 (Knoop scale), respectively.

carbon steel this property is less satisfactory. In salt-spray tests on steel coated with 0.0002, 0.0005, and 0.001 inch of electroless nickel, in comparison with similar panels coated with electrodeposited nickel, the protective value of the two types of coatings was virtually the same. However, the electroless cobalt does not compare so favorably with electrodeposited cobalt.

The yield or efficiency of the reduction based on the decomposition of hypophosphite is 37 and 66 percent for nickel and cobalt, respectively. About 2 grams of nickel or nearly 4 grams of cobalt are reduced by 10 grams of sodium hypophosphite. Because of the moderate yield and the present high cost of sodium hypophosphite, the process is expensive. Extensive commercial use of electroless plating is thus dependent upon a reduction in the price of this chemical.

## Test for Waterproofness of Leather

A new test for the waterproofness of leather, developed in the Bureau's leather laboratory, subjects the leather to conditions more comparable to those encountered in use than former methods and yields results in better accord with actual experience. Methods that have been previously used include measurements of the amount of water absorbed, the pressure required to force water through the leather, and the amount of water passing through the leather under a given pressure. None of these, however, is a direct measure of the waterproofness of leather and leather goods such as shoes that are ordinarily not subjected to high pressures of water or to immersion for any considerable length of time. Moreover, certain indications revealed by these tests do not agree with actual practice.

In the new test, leather specimens kept at normal water pressure are continuously flexed by a specially designed machine and at the same time are covered with water. The time, or the number of cycles, required for water to penetrate a specimen is taken as the measure of its waterproofness. A slight variation of the test, the addition of dyes to the water, has permitted the course of the water to be traced, thus revealing the mechanism of water penetration. This is seen to occur through hair follicles at points of stress along creases formed as the leather is flexed.

A large number of commercial leathers, including shoe-upper leathers, have been tested by the new method, and at the same time several of the factors that influence water penetration have been investigated. Among these factors are sampling, temperature, thickness, direction of penetration, and waterproofing agents.

The studies show that leather in general has a low resistance to water penetration, but that flesh-out leathers usually exhibit higher resistance than grain-out leathers. Commercial waterproofing materials do not have an appreciable effect on water resistance. Incorporation of large amounts of grease, however, will improve the water resistance, although this effect is decreased at lower temperatures.

## Flow Calorimeter for Specific Heats of Gases

A new flow calorimeter for more accurate measurements of the specific heats of gases has been developed by R. B. Scott of the Bureau's cryogenics laboratory. This apparatus was constructed in connection with a program sponsored jointly by the Bureau and the Office of Rubber Reserve for determination of the thermodynamic properties of materials involved in production of synthetic rubber and related substances. The greater precision of the new instrument is chiefly due to the almost complete elimination of heat leaks by various features of design. The small heat capacity of the

calorimeter also results in improved accuracy through the easier and more rapid attainment of a constant temperature distribution within the system, because only under conditions of steady temperature is an accurate measurement possible.

Accurate data on specific heats and other thermal properties of gases are of value to chemical industry, where they are used in calculations that predict the results of chemical reactions. With sufficient knowledge of the thermodynamic properties of the compounds involved, it is possible to state the range of temperature



within which a given reaction will occur and to estimate closely the relative amounts of the products.

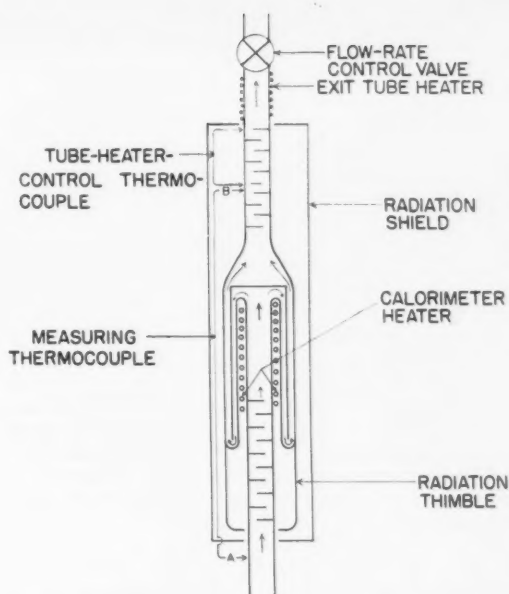
Specific heat is most simply obtained experimentally by adding a measured amount of heat to a fixed mass and recording the temperature rise. However, the heat capacity per unit volume of a gas is so low that small accidental heat leaks are often comparable in size to the heat used in actually raising the temperature of the gas. Reduction of the ratio of heat loss or gain to heat input is thus one of the chief problems of gas calorimetry. In the calorimeter constructed at the Bureau, this is accomplished by causing the gas to flow continuously at a uniform rate through an insulated chamber in which it is heated electrically. The heat capacity is then calculated from the heat input, the temperature rise of the gas, and the rate of flow. With gas calorimeters of this general type, a correction is usually made for heat leaks by making measurements at varying flow rates and extrapolating the apparent specific heats to infinite rate of flow, where such errors are negligible. In the new apparatus, however, special precautions have reduced heat leaks to such an extent that a correction for them is unnecessary. This advantage greatly lessens the number of necessary observations and also makes the results more trustworthy, as there are errors in calorimetric measurements that increase with increasing flow rate and cannot be removed by extrapolation to infinite flow.

The entire calorimeter is immersed in a constant-temperature bath. The gas whose specific heat is to be measured enters the system through a copper helix, in which it is brought to the temperature of the bath. It then flows into the calorimeter proper, where it traverses a labyrinth surrounding a constant heater, and leaves the calorimeter through a throttle valve. The temperature rise within the calorimeter is determined by appropriately located thermocouple junctions.

A uniform rate of gas flow is obtained by maintaining a constant pressure on the inlet side of the calorimeter and a constant lower pressure at the outlet. Adjustment of the throttle valve in the outlet tube then provides the desired rate of flow, which is determined by weighing the gas collected within a given time.

In the construction of the calorimeter, resistance to flow was reduced as much as possible, so that gases having low vapor pressures could be passed through the system at satisfactory rates. As it was necessary to measure the specific heats of vapors that corrode copper and silver, all parts traversed by the gas were made of monel metal.

Heat leaks to or from the interior of the calorimeter are minimized in several ways. Conduction along the inlet and outlet tubes is reduced through the use of tubing of monel, a poor-conducting metal, having a wall thickness of 0.01 inch. Radiation along the tubes is prevented by a series of baffles, which in addition promote thermal equilibrium between the gas and the tube walls. Heat leaks due to either conduction or radiation are also lessened by providing a long section of tubing between the inlet and the heater, and by heating the outlet to the final temperature of the gas. Radiation to or from the inner portion of the calorimeter is pre-



The gas whose specific heat is measured flows upward past a series of baffles, through a labyrinth about the calorimeter heater, and out the flow-rate control valve. Temperature rise of the gas is indicated by thermocouple junctions at A and B.

vented by a copper radiation shield kept at the temperature of the inner adjacent calorimeter wall by means of an electric heater. Necessary adjustments in the power to the heater are made in accordance with the readings of a three-junction thermocouple.

The accuracy of the apparatus was checked by measuring the specific heat of oxygen,<sup>2</sup> which had previously been accurately calculated at the Bureau from the latest spectroscopic data for this gas. The experimental values obtained at  $-30^{\circ}$ ,  $40^{\circ}$ , and  $90^{\circ}$  C differed from the spectroscopic values by 0.06, 0.00, and 0.10 percent, respectively. From a consideration of the data for oxygen, the probable error of the specific heats obtained with this calorimeter is estimated as 0.07 percent.

The new calorimeter has been used at the Bureau by Ruth K. Cheney, Jane W. Mellors, R. B. Scott, and P. F. Wacker to determine the specific heats from  $-30^{\circ}$  to  $90^{\circ}$  C of isobutane, isobutylene, 1-butene, *cis*-2-butene, butadiene, styrene, and ethylbenzene.<sup>2,3</sup> Isobutane is a principal raw material for the production of aviation gasoline and is also converted to isobutene—a substance used in large quantities for the preparation of butyl rubber and isobutanol. 1-Butene, like the 2-butenes, is converted to butadiene, the major component of GR-S rubber. A knowledge of the thermodynamic properties of these materials is expected to aid in the more efficient operation of plants in which they are produced or used.

<sup>2</sup> Paul F. Wacker, Ruth K. Cheney, and Russell B. Scott, J. Research NBS **38**, 651 (1937) RP1801.

<sup>3</sup> Russell B. Scott and Jane W. Mellors, J. Research NBS **34**, 243 (1935) RP1640.

# Electron Tube Research

A radio tube smaller than the eraser of a lead pencil—only a trifle larger than a grain of rice—has been developed by the electron tube laboratory of the National Bureau of Standards. This “rice-grain” tube, known as the “microtube,” is but one development of a basic and applied research program on vacuum tubes, undertaken by the laboratory in collaboration with industry, for military and industrial uses.

The various phases of the Bureau's fundamental and applied tube research are of great importance to the science of electronics. An extensive study of electron emission from cathodes and other elements in the tube envelope is expected to lead to the eventual production of tubes more dependable, longer-lived, and perhaps cheaper than those now available. Other research deals with the prevention of “gas clean-up,” the gradual absorption of gas in high-current relay and rectifier tubes used in industry. Finding and eliminating the causes of mechanical tube noise (“microphonics”) constitutes another important phase of the electron tube laboratory's work, as well as the development, improvement, comparison, and standardization of test methods and equipment for evaluating tube performance.

The most significant contribution made by the electron tube laboratory to the field of electronics is the development of new types of subminiature tubes in cooperation with private industry. In contrast to prewar tubes created by industry primarily for use as amplifiers in hearing aids, the new subminiature tubes are suited to a variety of other applications, such as detectors, amplifiers, rectifiers, and oscillators. In the course of this work, Bureau scientists found that a tube  $\frac{1}{4}$  inch by 1 inch would perform specific functions just as well when simplified in certain ways. Simplification made further reduction in size possible, and thus, by a process of evolution, the tiny microtube was developed. However, further details on this tube, which has various military applications, cannot be disclosed at this time.

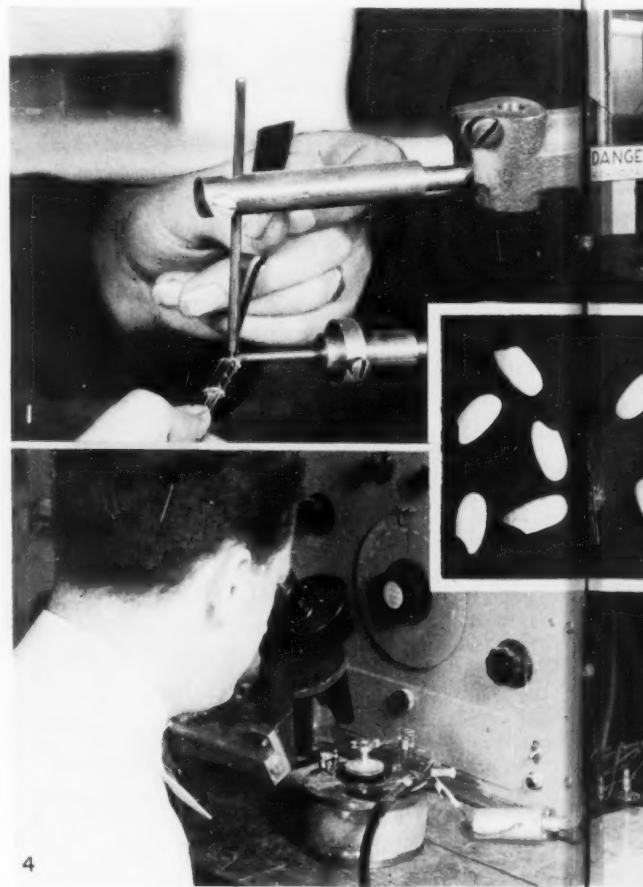
Although the Bureau participated actively in all this work, one of its particular accomplishments is the reduction of microphonics and internal tube noise. Subminiature tubes, in conjunction with printed circuitry, offer important aids to the entire electronics art. In fact, it is just such a tube, with a coil and other components printed on its envelope, that made possible the miniature radio transmitting and receiving sets described in a recent issue of the Bulletin.<sup>4</sup>

Subminiature tubes, having proved themselves, are now considered a new tool of great potential value. Manufacturers are accordingly devoting much time and effort to seeking ways and means of exploiting the possibilities of such tubes in radio sets, improved hearing aids, and other applications.

Typical of other developments that will eventually have a profound effect in the industrial and commercial fields are the proposed long-life tubes that are being developed for use in electronic computing machines.

Older models of such machines have utilized as many as 18,000 tubes, and some of the newer models, as well as others now contemplated, require as many as 2,000 for a single machine. It is of paramount importance, therefore, to reduce tube failures to a minimum, thus not only maintaining continuity of service but also greatly simplifying the problem of trouble-shooting and tube replacement. These new tubes are expected to have a life of 15,000 to 20,000 hours, or ten to twenty times the expected life of present-day computer tubes. They are being developed by a tube manufacturer working under a development contract.

In an effort to solve one of the most puzzling problems encountered in the fabrication and operation of vacuum tubes, the Bureau, through its electron tube laboratory, is now cooperating actively with industry in a study of impurities in cathode structures. It has been known



Electric spot welding (1) of tiny elements is one of the methods used in the fabrication of the microtube. (2) Following evacuation and sealing, the microtube is inspected. (3) Stroboscopic illumination of completed tubes permits stroboscopic comparison of the microtube with rice grains (3) illustrates the minute size of the microtube.

<sup>4</sup> NBS Technical News Bulletin 34, No. 5 (May 1947).

for some time that small amounts of impurities in the base nickel, such as iron, carbon, silicon, and manganese, affect the emissivity of an oxide-coated cathode in ways not yet completely understood. This matter was considered so important that the American Society for Testing Materials created a special committee with industry-wide representation to obtain, if possible, a quantitative answer as to the effects contributed by any particular impurity or combination of impurities. The tube manufacturers have deemed it wise, for the most part, to approach this problem from a statistical point of view, hoping to obtain satisfactory data from a large amount of sampling.

The method proposed by the electron tube laboratory aims to reduce experimental variables to a minimum. This method is one of synthesis, in which carefully controlled amounts of the different impurities will be deposited, singly or in combination, directly in or upon the base nickel, thus eliminating from the investigation many of the variable factors. Preliminary studies and

experiments have confirmed the feasibility of this procedure, and the investigation is being intensified.

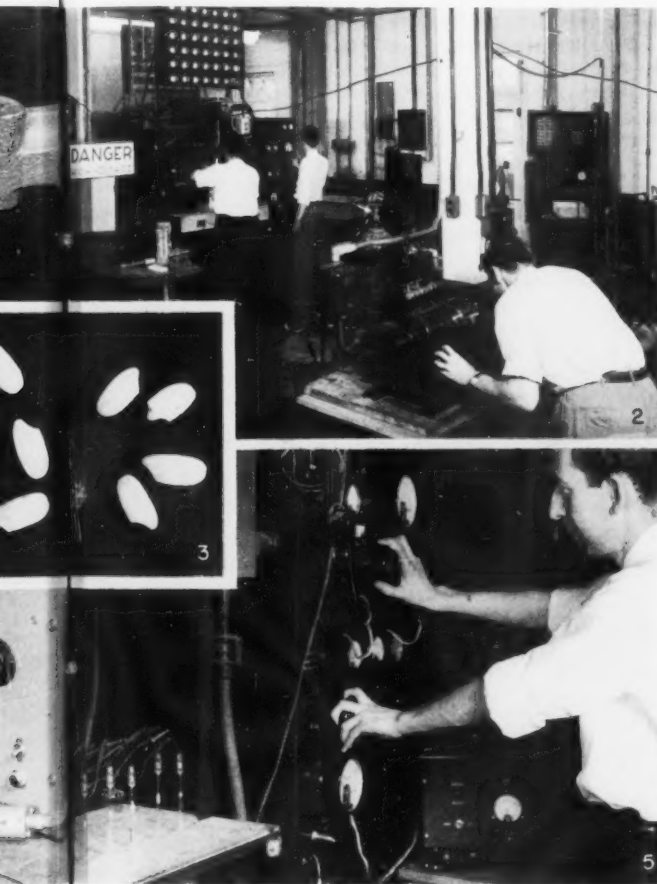
Another typical problem relating to applied research is the present intensive study of the phenomenon known as "gas clean-up," which refers to the mystifying disappearance of gas from a gas-filled tube. The aim in this particular case is to obtain comprehensive information on the factors involved, so that gas disappearance can be minimized or utilized in future tube designs and applications. The investigation thus far indicates the anode as the probable recipient of the gas. It is thought that the gas ions strike the anode surface with sufficient force to penetrate the surface and remain lodged within the material. The release of this gas from the anode can then be effected only by heating the latter to a high temperature.

The disappearance of gas at 3 to 10 microns pressure from a tube in just a few hours, under certain operating conditions, illustrates the extent of this phenomenon. If a way can be found to prevent gas-clean-up, it will then be possible to construct satisfactory low pressure tubes. Low order pressures of 3 to 10 microns would permit high inverse voltage break-down ratings—highly desirable but impossible of attainment today. Such an achievement, therefore, would represent a major advance in tube development.

The elimination of the phenomenon of "microphonics", which is due to vibration of the internal structure of the tube, and which is therefore present to a greater or lesser degree in every vacuum tube, would solve many of the problems now confronting the manufacturers of electronic equipment. Incredibly small variations in the internal structure of the vacuum tube will often produce very great changes in operating characteristics. In addition, such a structure is inherently subject to many directions and amplitudes of vibration. These factors make the entire subject of microphonics an extremely complicated one. The electron tube laboratory has already achieved important progress in the field of microphonics, but thus far the work has pertained only to secret requirements of the armed forces.

In order to keep abreast of all independent developments in electron tube science, the Bureau maintains close contact with the industry. The information thus obtained is utilized in establishing and maintaining extensive data on the characteristics of most commercial tubes. This collected information is invaluable in carrying on the consulting service that the Bureau renders to industry and to other government agencies. The tests that the laboratory performs on new tubes and on tubes of particular interest to application engineers provide an additional source of technical data.

The laboratory has direct representation on military and civilian committees concerned with various aspects of research, development, and standardization. It is also represented on the American Standards Association Committee on Electron Tubes, and on the Subcommittee of the American Society for Testing Materials, which is conducting the investigation of cathode structures. Committee work for the armed forces is carried on through representation on the Panel on Electron



of the delicate operations in assembly of experimental subminiature tubes. Following evacuation, the tubes are tested electrically (5) before final tests. The tests determine the tubes' ability to withstand vibration and shock. The tests indicate the trend in the trend toward miniaturization of vacuum tubes.

Tubes, and on certain of its subpanels, which report to the Joint Research and Development Board, the coordinating body of the Armed Services for research and development.

The electron tube laboratory is one of the newest important units of the National Bureau of Standards. Staffed with a small group of highly trained physicists and technicians and equipped with the most modern equipment available for small-scale electron tube design, manufacture and testing, its special tube studies benefit both industry and the consumer. Work on tubes

whose applications are primarily military is carried on directly for the Armed Services, often with the cooperation of industry. In addition to its research and development activities, the laboratory will test tubes for the various civilian agencies of the Government to insure compliance with purchase specifications. To the other sections of the Bureau, the laboratory provides such important services as tube repairs, the duplication of special tubes, and expert advice on tube problems, which frequently require new methods of approach or the development of new techniques.

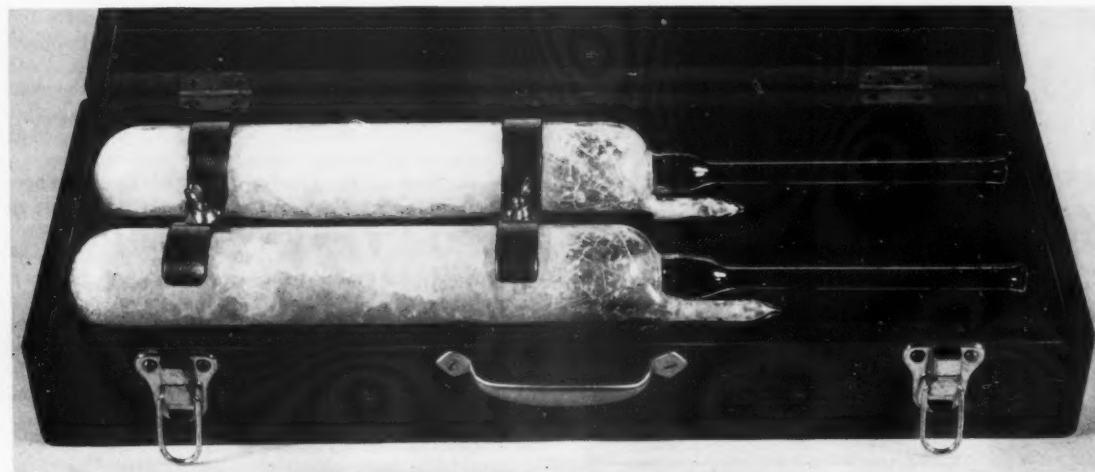
## Benzoic Acid Cells Provides Thermometric Standard

A new thermometric standard utilizing the freezing point of benzoic acid as a fixed temperature has been developed and made available to industrial and other laboratories by the Bureau. The acid, which is specially purified for the purpose, is contained in a cylindrical chamber, about 12 inches long and 2 inches in diameter, provided with a thermometer well. This device, known as a benzoic acid cell, affords a means of calibrating platinum resistance thermometers and thermocouples at a temperature near  $100^{\circ}\text{C}$  more rapidly and conveniently and, under certain conditions, more accurately than is possible with the boiling point of water. The temperature provided by the cell is virtually the triple point of benzoic acid ( $122.362^{\circ}\text{C}$ ).

The boiling point of water is well established as a fixed point in thermometry not only for historical reasons but also because the thermal properties of water and its ease of purification make it an almost ideal standard substance. The laborious barometric measurements required for accurate observations of the

steam point have constituted the principal drawback to the use of this temperature in thermometer calibration. Recently, however, as the result of an investigation of the properties of benzoic acid by F. W. Schwab and Edward Wichers of the Bureau, it has been found possible to use the freezing temperature of benzoic acid for this purpose with a precision comparable to that of the steam point as observed in most standardizing laboratories.

To use the cell, it is heated in an oven until the acid has melted and reached a temperature a few degrees above its freezing point. The cell is then shaken until the liquid begins to freeze, forming a mush of fine crystals; thereupon the cell is put in a Dewar flask to retard the rate of freezing. The resistance thermometer (or thermocouple) to be calibrated is placed in the well, and observations of resistance (or emf) are made for a period of 1 to 2 hours. After the first 30 minutes, the temperature of the cell is constant within  $0.001^{\circ}\text{C}$  for an hour or longer and is reproduced



The benzoic acid cell, consisting of a thermometer well surrounded by a chamber containing benzoic acid, utilizes the freezing temperature of the acid to provide a fixed point for calibration of platinum resistance thermometers and thermocouples.



in repeated "freezes" within a maximum range of 0.002 degree C.

The freezing temperature of each cell issued by the Bureau is certified to an accuracy of  $\pm 0.003$  degree C. However, this is an estimate of the uncertainty involved in fixing the temperature on the International Temperature Scale, and does not relate to the constancy with which the temperature is maintained in a given cell during the time necessary for observations or to the day-by-day reproducibility of the cell.

Certified cells<sup>3</sup> may be obtained from the Bureau for a fee of \$100.00 each. An uncalibrated companion

cell containing acid of lower purity is offered for \$15.00; it is useful for practice in manipulation and for prewarming of the Dewar flask. Cells are available with thermometer wells in three diameters, 8, 10, and 12 millimeters. The diameter that most nearly fits the instrument to be calibrated should be used.

<sup>3</sup> More detailed information about these cells can be obtained from the Bureau. Observations on the pertinent properties of benzoic acid are given in The freezing temperature of benzoic acid as a fixed point in thermometry, by Frank W. Schwab and Edward Wichers, J. Research NBS **24**, 332 (1945) RP1647. This paper has an appendix by Frank W. Schwab and E. R. Smith, which gives a convenient method of calculating temperatures from observations of the resistance of platinum thermometers. Reprints of the paper, with its appendix, are available from the Superintendent of Documents, Washington 25, D. C., at a cost of 10 cents.

## Precision of Telescope Pointing

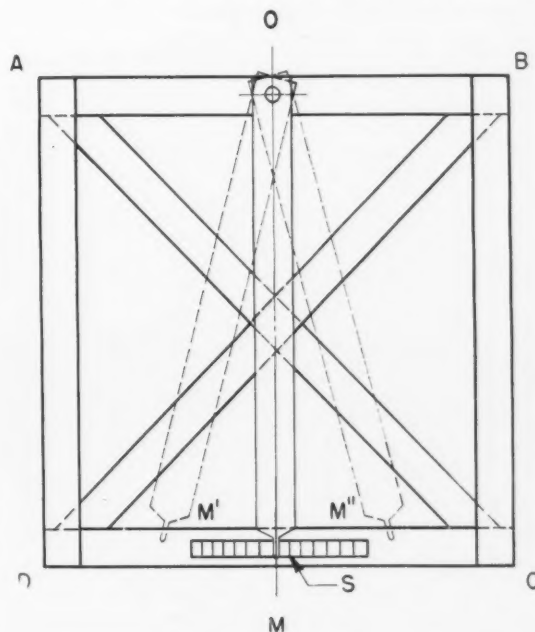
The accuracy and precision of measurements made with optical instruments—whether by the surveyor determining boundaries, the gunner locating a target, the astronomer mapping the stars, or the physicist in the laboratory—depend upon the errors introduced when sighting through the telescope portion of the instrument. These errors fall into two main classes. One large group of errors arises basically because of imperfections of the mechanical mounts by which the telescope is directed, errors of the graduated circles serving as accessories to the telescope, and improper kinaesthetic relations between the type of slow movement and the reactions of the observer. Errors of this class can continually be reduced as the art of the instrument designer improves. There is also a more subtle class of errors that arise because of the limitations of the observer's eye and the fact that observations are usually made through a column of air, a turbid medium that is optically imperfect. With this group of errors the instrument designer can do little, but such errors can be profitably studied in order to determine the choice of conditions that will render their effects least unfavorable.

The latter class of errors has been comprehensively investigated by Dr. Francis E. Washer and his associates of the optical instruments laboratory at the National Bureau of Standards. Their investigations fall generally into three classifications: (a) A study of the variation in the probable error of pointing as a function of the distance to the target. This is a direct evaluation of the manner in which the optical imperfections of the air affect the precision of pointing. (b) A measure of the probable error of pointing, as governed by the choice of magnification, both in the laboratory where the effect of the short column of air is unimportant and for long air paths. (c) An experiment designed to show that the difference in the precision achieved in laboratory and outdoor pointing is in fact due to the effect of the atmosphere.

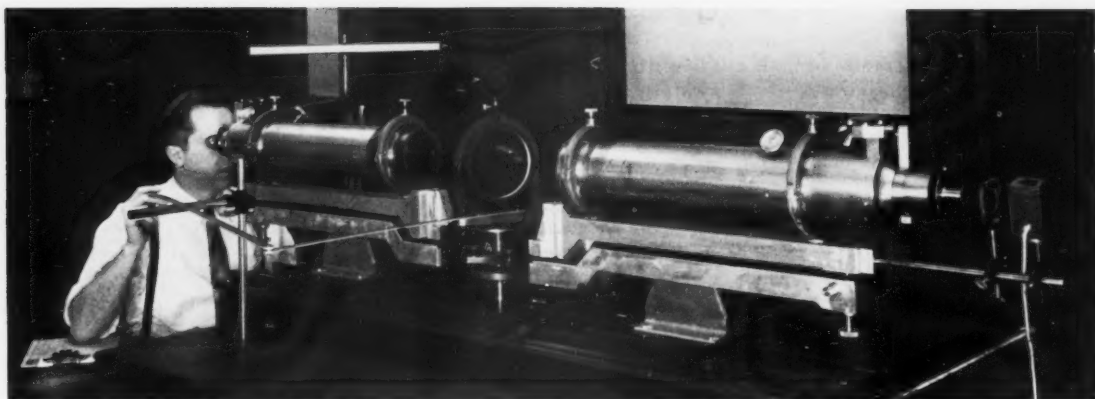
To eliminate the effects of mechanical error, the telescope is mounted rigidly and left undisturbed during the course of a series of observations. The image of the target is moved with respect to the cross hairs by means of a weak prism, placed in front of the objective and capable of rotation about its vertical axis. To

make an observation, the prism is oscillated about this axis causing the image of the target to sweep from right to left of the cross hairs. The amplitude of oscillation is successively decreased until the image of the target appears to coincide with the cross hairs in the telescope eyepiece. The position of the prism is then noted with an auxiliary telescope and scale. The probable error,  $PE_s$ , is determined from a series of ten such observations.

The quantity,  $PE_s$ , that is determined in these experiments for the combination of telescope and observer is the probable error of a single pointing about the instantaneous "true" pointing at the time at which it is made.



Diagrammatic sketch of target used to determine the accuracy of telescope pointing as affected by intervening air column. Arm OM is moved to coincidence with the center of the cross under direction of observer 4,710 meters away.



Arrangement of telescope, prism, and collimator in the Bureau's optical instruments laboratory for studying the effect of magnification on precision of telescope pointing for indoor targets. The prism (between collimator and objective) can be rotated about its vertical axis; thus the target image is moved to coincide with the cross lines of the eyepiece for successive settings. The probable error of a single pointing varies with magnification.

It is a measure of the error of a single pointing determined from a number of pointings taken rapidly, and does not contain any appreciable effect of drift.

To study the effect of range on the precision of pointing, 15 outdoor test objects were used. These objects, readily discernible from the laboratory, were at accurately known distances 100 to 13,600 meters from the point of observation. Several series of observations were made on each target by each of two observers for variety of outdoor weather conditions. For such outdoor pointing, no appreciable difference between observers was found. The probable errors fluctuate considerably from day to day, depending on weather conditions. However, when all values for each target were averaged, the following relation between the probable error,  $PE_s$  (expressed in seconds), and range,  $d$  (meters), was derived:

$$PE_s = \pm (0.064 d^{1/3} \pm 0.19).$$

For practical purposes, the variation of  $PE_s$  with range can be neglected and the value

$$PE_s = \pm (0.62 \pm 0.01)$$

taken as the average for all distances and weather conditions.

In some of the experiments, a red filter was used, but no improvement in precision resulted as compared with that attained without the filter. In addition, a peculiar effect indicating a real difference between right- and left-eye pointing was noted; that is, observers tend to point more to one side of the target with one eye than with the other. In some cases this effect, found also in indoor pointing, amounts to as much as 1 second.

As control of conditions for outdoor pointing is not practicable, and as experience has shown that low and consistent values of  $PE_s$  are obtained indoors, the study of the effect of magnification was made on indoor targets using a special arrangement of a telescope, prism, and collimator. For these experiments the magnification was varied from 6 to 414 diameters with the

brightness of the target background maintained at a level compatible with easy vision. The probable error of a single pointing,  $PE_s$ , is found to vary with the magnification,  $M$ , as indicated in the equation

$$PE_s = \frac{4.96}{M} + 0.07.$$

The value 4.96 seconds is the vernier acuity of the observer's eye and consequently different values for different observers may be anticipated. In fact, it is possible that an arrangement of this sort used under proper conditions might prove to be a convenient means for measuring the vernier acuity of the eye.

The values of  $PE_s$  found in the work with indoor targets are very much lower than are found for the same magnification with outdoor targets. It seems probable that turbulence of the air column intervening between target and observer is the chief cause of this discrepancy. This was corroborated by a study wherein the effect of the air column could be introduced or eliminated at will. To do this a special target was placed on the tower of the Soldiers' Home, 4,710 meters from the observer at the National Bureau of Standards. The target consisted of two diagonals intersecting to form a cross. A swinging arm fastened to the top of the target could be moved into coincidence with the center of the cross (under direction of the observer) and successive settings read with the aid of a scale located at the bottom of the target.

As the image-forming light from both the intersection of the diagonals and the swinging arm traversed the same column of air to arrive at the viewing telescope, and as the setting was made with no reference to the cross hairs in the telescope, the setting was considered to be independent of the air column. Following a series of observations of this kind, several settings were made by the observer at the telescope, using the weak prism and the cross hairs in the telescope. The values of  $PE_s$  found by the "air-column-eliminated" method are very much lower than those found by the "air-

column-present" method. They agree closely with the values for pointing with the indoor target at the same magnification.

Results of the investigations at the Bureau prove conclusively that the influence of the air column limits the ultimate precision obtained by telescope pointing for outdoor targets. From a study of data on pointing, it is evident that there is little gain in pointing accuracy as a result of increasing the magnification above 20 diameters, although high magnification may still be used to advantage for distinguishing detail.

### References

- F. E. Washer and Helen B. Williams, *J. Opt. Soc. Am.* **36**, 400 (1946).  
F. E. Washer and Helen B. Williams, *J. Research NBS* **35**, 479 (1946) RP 1717.  
F. E. Washer, *J. Research NBS* **39** (1947) RP 1820.  
F. E. Washer and L. W. Scott, *J. Research NBS* **39**, 297 (1947) RP 1829.

## Conference of State Utility Commission Engineers

The Twenty-Fifth Annual (Silver Anniversary) Conference of the State Utility Commission Engineers was held at Madison, Wisc., June 10 to 12, 1947, with John W. Kushing of the Michigan Public Service Commission, vice-chairman of the Conference, presiding. R. L. Lloyd, of the Bureau's Codes and Specifications Section, served as secretary of the Conference, an office normally held by a member of the Bureau staff. Fifty-one engineers attended as representatives of 20 States, the Province of Ontario, the National Association of Railroad and Utility Commissioners, and the Federal Government.

The program of technical papers included three by members of the Bureau staff. Two of these related to measurement of natural gases, the testing of rotary gas meters, by H. S. Bean of the gas measurements laboratory, and tests of instruments for determination

of the specific gravity of gases by J. H. Eisman of the gas chemistry laboratory. F. L. Hermach of the electrical instruments laboratory described principles and procedures used in the precise testing of watt-hour meters. Technical papers were also presented on production and use of propane gas, government power projects and taxes, the history of the telephone industry, the mobile telephone problem, electric rates, water rates, and other topics of importance to utility commission engineers.

The Policy Committee recommended that the National Bureau of Standards be considered for the 1948 annual meeting, as this Conference has not been held at the Bureau for several years.

## Patents on Powder Metallurgy

A comprehensive list of powder metallurgy patents—a valuable source of technical information on this new and undeveloped science—has been compiled following an extensive study of patent literature in connection with an investigation in the field of powder metallurgy at the Bureau. Representing more than a century of progress in this art, the information, which was obtained from a collection search of 2,253 patents and classified in related groups with a short abstract for each invention, has been made available as NBS Miscellaneous Publication M184, *United States Patents on Powder Metallurgy*, by Raymond E. Jager and Rolla E. Pollard.

Published technical literature in this field is widely scattered, making it difficult for metallurgists to keep abreast of developments in this rapidly growing science. For this reason, the Bureau's listing and analysis of the patent literature classified as to production, handling and working, alloying, and application, should prove useful to those engaged in research and development in powder metallurgy. The publication is available from the Superintendent of Documents, Washington 25, D. C., at 30 cents a copy.

## Supplement to Directory of Specifications

A Supplement<sup>a</sup> to the National Directory of Commodity Specifications has been announced by the National Bureau of Standards. The 1945 edition of the Directory with this Supplement provides a complete listing, by name, designating number, and issuing or sponsoring organization, of all the standards, specifications, and methods of test in general use for commodities produced in or purchased by this country. These publications, which were prepared to meet the growing demand for an up-to-date authoritative compilation of such data, also include many items that are not strictly commodity specifications but closely related information essential to most users. Each specification, of which there are 44,000, is briefly summarized as to technical characteristics, scope, and special applications.

It has become widely recognized, especially by large organizations and purchasing agents, that the most logical way to achieve economy in purchasing is

through the use of dependable specifications. Only by means of a well formulated specification containing definite, concise, and complete statements concerning a product, can the buyer accurately present his requirements to the seller. Purchasing by specifications thus provides a workable system whereby both production and distribution can be coordinated to their mutual benefit in supplying the consumer.

The material in the Directory and Supplement is especially coordinated for the use of new manufacturers who need detailed information concerning finished goods, or practical data on available materials. It is equally valuable to old, established manufacturers who desire to introduce new products or to use materials with which they are not familiar.

<sup>a</sup> Prepared by Paul A. Cooley of the Bureau's Division of Codes and Specifications. Copies of the Directory (Miscellaneous Publication M178) and its Supplement may be obtained for \$4.00 and \$2.25, respectively, from the Superintendent of Documents, Government Printing Office, Washington 25, D. C.

The same convenient decimal system of commodity classification used in the Directory has been carried out in the Supplement, with some additions to take care of new materials. This system aids bodies that formulate specifications by informing them concerning previous standards, thus avoiding unnecessary duplication.

## Motion Picture on Dental Silicate Cement

A new sound film in color entitled "Silicate Cement" has been completed at the National Bureau of Standards, according to a joint announcement by the Bureau and Dr. M. D. Huff, Chairman of the Research Commission of the American Dental Association.

Since 1928 the National Bureau of Standards and the American Dental Association have conducted cooperative research on the physical and chemical properties of dental materials and the proper technics involved in their use. The present film, the first of a series that will stress the clinical significance of physical properties and the importance of technic, is a result of this research.

"Silicate Cement" demonstrates two cardinal prin-

cles that must be followed to get good restorations. The first is to incorporate as much powder as possible into a given quantity of liquid as rapidly as possible. The second is to protect the cement from loss or gain of water before mixing, during mixing, and throughout the hardening period. The effect of technic on such critical cement properties as setting time, strength, shrinkage, solubility, and staining is shown in a series of simple and vivid laboratory tests.

The picture, a 16-millimeter film photographed in color, has a running time of 18½ minutes. Information on the loan or purchase of this film can be received by writing to the Director, National Bureau of Standards, Washington 25, D. C.

The picture, a 16-millimeter film photographed in color, has a running time of 18½ minutes. Information on the loan or purchase of this film can be received by writing to the Director, National Bureau of Standards, Washington 25, D. C.

## NBS Publications

### Periodicals <sup>7</sup>

Journal of Research of the National Bureau of Standards, volume **39**, number 3, September 1947. (RP1823 to RP1828, inclusive).

Technical News Bulletin, volume **31**, number 9, September 1947, 10 cents.

CRPL-D37. Basic Radio Propagation Predictions for December 1947. Three months in advance. Issued September 1947, 15 cents.

### Nonperiodical

#### RESEARCH PAPERS <sup>7,8</sup>

RP1816. Absorption spectra in the detection of chemical changes in cellulose and cellulose derivatives. John W. Rowen, Charles M. Hunt, and Earle K. Plyler. Price 10 cents.

RP1817. Anodic current efficiency in the counterflow electrolysis of uranyl chloride solutions. Walter J. Hamer. Price 10 cents.

RP1818. Analysis by the mass spectrometer of a liquefied hydrocarbon containing C<sub>8</sub>-C<sub>10</sub> paraffins and olefins. Vernon H. Dibeler and Fred L. Mohler. Price 10 cents.

RP1819. An indentation method for measuring wear. Samuel A. McKee. Price 10 cents.

RP1820. Effect of magnification on the precision of indoor telescope pointing. Francis E. Washer. Price 10 cents.

RP1821. Heats of formation and isomerization of the eight C<sub>12</sub> alkylcyclohexanes in the liquid and gaseous states. Edward J. Prosen, Walter H. Johnson, and Frederick D. Rossini. Price 5 cents.

RP1822. Some recent advances in our understanding of the chemistry of portland cement. Robert H. Bogue. Price 10 cents.

#### SIMPLIFIED PRACTICE RECOMMENDATIONS <sup>7</sup>

R86-47. Surgical Gauze. (Supersedes R86-42). Price 5 cents.

R133-47. Surgical Dressings. (Supersedes R133-38). Price 10 cents.

R227-47. Plumbing Fixture Fittings and Trim for Housing. Price 5 cents.

#### MISCELLANEOUS <sup>7</sup>

MI84. United States Patents on Powder Metallurgy. Price 30 cents.

#### LETTER CIRCULARS <sup>9</sup>

LC873. Enamels, Publications by members of the staff of the National Bureau of Standards, together with a list of Federal Specifications. (Supersedes LC587D).

LC874. Radio publications by the staff of the National Bureau of Standards. (Supersedes LC781.)

LC875. Technical radio broadcast services, radio station WWV.

### Articles by Bureau Staff Members in Outside Publications <sup>10</sup>

Electronics and the future. E. U. Condon. Electrical Engineering (33 West 39th Street, New York 18, N. Y.) **66**, 355 (April 1947).

Instruments for flight testing airplanes. W. G. Brombacher. Instruments (1117 Wolfendale Street, Pittsburgh 12, Pa.), **20**, 700 (August 1947).

Simplified head for laboratory fractionating columns. Frank L. Howard. Analytical Chemistry (332 West Forty-Second Street, New York 18, N. Y.) **19**, 144 (1947).

<sup>7</sup> Send orders for publications under this heading only to the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Annual subscription rates: Journal of Research, \$4.50 (foreign \$5.50); Technical News Bulletin, \$1.00 (foreign \$1.35); Basic Radio Propagation Predictions, \$1.50 (foreign \$2.00). Single copy prices of publications are indicated in the lists.

<sup>8</sup> Reprints from August Journal of Research.

<sup>9</sup> Available on request from the National Bureau of Standards, Washington 25, D. C. Letter Circulars are prepared to answer specific inquiries addressed to the Bureau, and are sent only on request to persons having a definite need for the information. The Bureau cannot undertake to supply lists or complete sets of Letter Circulars or send copies automatically as issued.

<sup>10</sup> These publications are not available from the Government. Requests should be sent to the publishers.



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